CLAIMS

1. A process for preparing a compound of formula

in which R represents linear or branched C_1 - C_5 aliphatic acyl or benzoyl, optionally substituted with C_1 - C_5 alkyls, C_1 - C_5 alkoxyls or halogens,

which comprises the reaction of coupling of a compound of formula

in which

15 R represents a linear or branched C_1 - C_5 aliphatic acyl or benzoyl, optionally substituted with C_1 - C_5 alkyls, C_1 - C_5 alkoxyls or halogens,

R' represents R or a linear or branched C₁-C₅ alkyl,

with a compound of formula

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in which R", being identical or different, represents a C₁-C₆ alkyl or a phenyl, in the presence of a Lewis acid and in an inert organic solvent, characterized in that said Lewis acid is added at a temperature below 0°C.

- A process according to claim 1 in which said addition of catalyst is carried out at a temperature below –10°C, preferably between approx. –15 and –20°C.
 - 3. A process according to claim 1 in which, on completion of said addition of catalyst, the reaction mixture is held further at the same temperature.

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- 4. A process according to claim 1 in which R and R' represent acyl, preferably acetyl, and R" represents methyl.
- 5. A process according to claim 1 in which said Lewis acid is selected from trimethylsilyltrifluoromethanesulphonate and tin tetrachloride, and is preferably tin tetrachloride.
 - 6. A process according to claim 1 in which said inert organic solvent is selected from chlorinated solvents or aromatic solvents, preferably chlorinated solvents.

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- 7. A process according to claim 1 in which said compound of formula II, in which R has the meanings stated above, is further submitted to a reaction of deprotection to give doxifluridine of formula I.
- 25 8. A process for the preparation of doxifluridine of formula

that comprises a process according to one of the claims from 1 to 7.